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OIL-RESISTANT ELASTOMERS WITH LOW PERMEABILITY TO NITROGEN

John A. Williams

Army Weapons Command
Rock Island, Illinois

August 1972

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TECHNICAL REPORT

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RESEARCH DIRECTORATE

WEAPONS LABORATORY USAWECOM

RESEARCH, DEVELOPMENT AND ENGINEERING DIRECTORATE

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The purpose of this work at the Research Directorate, Weapons Laboratory, WECOM, was to reduce the nitrogen gas transmission rate (GTR) and permeability coefficient of oil-resistant elastomers without adversely affecting other physical properties. This was accomplished through the use of various fillers of proper size and shape to produce a blocking effect, thus reducing gas transmission through the polymer film. Carbon black and most nonblack fillers produced only a small reduction in GTR, whereas platelike mica and graphite fillers produced significant reductions of as much as 78 per cent. These reductions in GTR were achieved without serious loss of oil resistance or compression set; but, in some cases, loss in flexibility at low temperatures was noted. (U) (John A. Williams)

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OBJECTIVE

The purpose of this work was to develop elastomeric compounds with low permeability to gases for potential use in the fabrication of weapon components such as accumulator bladders for recoil mechanisms, fuel pump diaphragms, and moistureproof barrier bags for combustible-cased ammunition. This work was conducted by personnel of the Research Directorate, Weapons Laboratory, WECOM.

BACKGROUND

The resistance of elastomers to the penetration of gases, vapors, and liquids is an important factor in their use in weapon components. Whether high or low resistance to the penetration of gases and liquids is desired is dependent upon the application of the material. In tire inner tubes, for example, low permeability to air is desired; whereas, in water purification systems, high permeability to water and resistance to the permeation of other substances is needed.

The development and the utilization of low-permeable elastomeric compounds are important in the development of weapon components. Barrier bags having low water-vapor transmission rates could mean the difference between success or failure of those weapon systems in which combustible-cased ammunition is used. Elastomers with low permeability to combustible liquids would mean a big step forward in the development of the liquid propellant gun system.

High resistance to the penetration of gases and liquids with good low-temperature flexibility and good oil and fuel resistance are usually not found in the same elastomer.[1] Yet these requirements are often necessary for the proper function of weapon components and accessories such as seals, O rings, diaphragms, bladders, and fuel storage tanks. For example, the need exists for rubber accumulator bladders in recoil mechanisms to prevent nitrogen/hydraulic fluid interchange. These and other requirements for low-permeable elastomers indicate the need for the work described herein.

New polymeric materials are becoming available at a rapid rate, and their usefulness in weapon components must be determined. The best way to acquire knowledge of the engineering potential of these new materials is through test and evaluation. Information obtained in this work will prove invaluable in the development of future weapon components.

APPROACH

In previous work on the permeability of elastomers, efforts were focused on the effect of polymer structure on the diffusion rates of gases and liquids.[2] In this work, the effect of various types of fillers and plasticizers on the nitrogen gas transmission rate of oil-resistant elastomers was determined. The effect of these compounding

ingredients on the inherently good oil-resistance and the low-temperature properties of the elastomers was also determined.

Elastomers and compounding ingredients were mixed on a 6 by 13 inch open-roll mill. Standard test pads of butadiene-acrylonitrile and polyepichlorohydrin rubber were cured for 30 minutes at 310°F and 347°F, respectively. Fluorosilicone test pads were mold-cured five minutes at 240°F and air oven postcured for eight hours at 392°F. All physical properties were determined with the use of ASTM methods. Nitrogen gas transmission rate and permeability coefficient were determined by ASTM Method D1434-66, Method V, on test pads 0.030 inch in thickness.[3]

RESULTS AND DISCUSSION

Gas Transmission Rate (GTR), as determined by the volumetric method, is a measure of the volume of gas passing through a specific cross-sectional area of material. The thickness of the material is not included in this calculation and must be stated separately. Permeability coefficient is also a measure of the permeability of a material; but, in this measure, the thickness of the test specimen is included in the calculation. In determining GTR, the temperature of the specimen and the gas must be constant during the test period. The gas pressure difference across the specimen must also be kept constant. In this work, the temperature was maintained at 77°F and the pressure difference at 48 psig.

Four classes of oil-resistant elastomers were chosen for this study; namely, fluorosilicone, low and high acrylonitrile content butadiene/acrylonitrile copolymers (NBR), and polyepichlorohydrins.[4] Of all known elastomers, the fluorosilicones possess the best combination of resistance to deterioration by oils, fuels, and other fluids, coupled with excellent flexibility at -60°F and nonbrittleness at -67°F. This class of elastomer is, however, somewhat deficient in strength and resistance to abrasion. The low acrylonitrile NBR polymers are not as resistant to fluids as are the fluorosilicones nor do they function at temperatures as low as the fluorosilicones, but they have good strength. The high acrylonitrile content NBR elastomers have excellent resistance to fluids and very high strength, but are incapable of functioning below about 25°F. The polyepichlorohydrins are recently developed elastomers which exhibit a good balance of fluid resistance, strength, and low-temperature performance. The purpose of this study was to reduce the permeability to gases of these four types of elastomers by reduction of the number and size of the molecular voids in the elastomers. This was to be accomplished through the use of large particle-size fillers and by the increasing of the density of the cross links in the vulcanizates. Because of the insufficient time allowed only a limited study of the effects of cross-link density was possible.

Fluorosilicone rubber is well known for its poor resistance to nitrogen permeation. Table I shows the GTR of a 30 mil film to be 2865.60 cm^3/24 hrs/$meter^2$/atmosphere, which is very high. The addition

of 30 pphr of 325 mesh mica, a platelike filler, produced a blocking action to the passage of the nitrogen which reduced the GTR by 55 per cent. The addition of this filler did not significantly affect other physical properties.

Table II shows the physical properties of a low acrylonitrile content butadiene-acrylonitrile rubber. This rubber has a low nitrogen GTR of 97.67 cm^3/24 hrs/meter^2/atmosphere. The reason for the low GTR is that the large nitrile groups attached to the polymer chain blocked the passage of nitrogen molecules. The addition of 325 mesh mica and graphite increased this blocking effect and reduced GTR by 70 and 77 per cent, respectively. Magnesium silicate, Dixie clay, and MT carbon black were less effective in reducing permeability. These fillers did not significantly affect the other physical properties except the low temperature flexibility, which was raised an average of 13°F.

Table III indicates that the addition of a plasticizer (TP-95, di(butoxy-ethoxy-ethyl) adipate) increases the nitrogen GTR by 100 per cent. The belief is that this increase can be attributed to the nitrogen molecules having a more friction-free path through the polymer chains. The addition of 325 mesh mica and graphite reduced the GTR of this plasticized rubber compound by 78 and 74 per cent, respectively. The other fillers in this table substantially reduced permeability but by a lower percentage. The use of this plasticizer lowered the useful low-temperature range of this vulcanizate by about 25°F. Compression set is adversely affected by these filler/plasticizer additions and, in one compound, is 300 per cent higher.

Table IV shows the physical properties of a high acrylonitrile content butadiene-acrylonitrile rubber. This rubber has more nitrile groups attached to the polymer chain than the low acrylonitrile content rubber shown in Tables II and III. This greater number of nitrile groups blocking the passage of nitrogen produced a very low GTR of 12.54 cm^3/24 hrs/meter^2/atmosphere. The addition of the graphite reduced the GTR 30 per cent, but the 325 mesh mica produced only a 9 per cent decrease. Because of the high viscosity of this rubber, the belief is that the mica is ground into a smaller particle size during the mill mixing of this compound. These smaller particles offer less blocking action to the nitrogen resulting in a higher GTR than would be expected. The other fillers did not reduce gas permeability.

Table V shows the physical properties of a polyepichlorohydrin rubber. This rubber demonstrates a very low nitrogen GTR of 11.17 cm^3/24 hrs/meter^2/atmosphere. The belief is that the large chlorine atoms attached to the polymer chain block the path of the nitrogen through the rubber film. The addition of 325 mesh mica reduced this low GTR another 35 per cent to 7.26 cm^3/24 hrs/meter^2/atmosphere. Other fillers were less effective in reducing GTR. The oil resistance remained excellent, but other physical properties were adversely affected by these fillers.

Table VI shows the physical properties of an epichlorohydrin-ethylene oxide copolymer. This rubber has a very low GTR of 31.40 cm^3/24 hrs/$meter^2$/atmosphere. In this series of compounds, the filler additions had more affect on the GTR than was expected as is shown in the results obtained from other elastomers tested. Graphite and 50 mesh mica reduced GTR 63 and 61 per cent, respectively. The other fillers also significantly reduced GTR without seriously affecting other physical properties.

Table VII shows the physical properties of a copolymer of epichlorohydrin and ethylene oxide with the addition of three types of plasticizers. Diisoamyl adipate lowered the useful low-temperature range of this elastomer 15°F, but the nitrogen GTR was increased by 200 per cent.

TABLE I

EFFECT OF 325 MESH MICA ON THE NITROGEN GAS PERMEABILITY OF FLUOROSILICONE RUBBER

	Parts by Weight	
Compounding Ingredients		
Silastic LS63U	100	100
Ferric Oxide	3	3
Cadox TS-50	1.3	1.3
325 Mesh Mica	--	30
Physical Properties		
Tensile Strength, psi	1170	890
Modulus @ 200%E, psi	600	870
Elongation, %	315	215
Hardness, Shore A	56	70
Permeability to Nitrogen, Tested at 77°F and 48 psig		
Thickness (mils)	30	30
Gas Transmission Rate, cm^3/24 hrs/$meter^2$/atmosphere	2865	1275
Permeability Coefficient in Barrers	33.3	14.8
70 hrs/212°F/ASTM #3 Oil		
Tensile Strength, psi	820	710
Modulus @ 200%E, psi	470	620
Elongation, %	260	215
Hardness, Shore A	54	64
Volume Change, %	+10	+25
Compression Set, Method B, 70 hrs/212°F, %	17	36
Low Temperature Flexibility, ASTM D1043, Temp. where Young's Modulus is 10,000 psi, °F	-70	-62

TABLE II

EFFEC ILLERS ON THE NITROGEN GAS PERMEABILITY OF A LOW ACRYLONITRILE CONTENT BUTADIENE-ACRYLONITRILE RUBBER

	Parts by Weight					
Compounding Ingredients						
Paracril AJ	100	100	100	100	100	100
Stearic Acid	1	1	1	1	1	1
Zinc Oxide	5	5	5	5	5	5
Santocure	1	1	1	1	1	1
Sulfasan R	1.3	1.3	1.3	1.3	1.3	1.3
Methyl Tuads	1.3	1.3	1.3	1.3	1.3	1.3
FEF Carbon Black	50	50	50	50	50	50
325 Mesh Mica	--	50	--	--	--	--
Graphite	--	--	50	--	--	--
Magnesium Silicate	--	--	--	50	--	--
Dixie Clay	--	--	--	--	50	--
MT Carbon Black	--	--	--	--	--	50
Physical Properties						
Tensile Strength, psi	2240	2140	1680	2390	2030	2080
Modulus @ 200%E	1180	1240	1480	1130	1340	1460
Elongation, %	315	310	220	325	340	240
Hardness, Shore A	80	81	81	85	80	78
Permeability to Nitrogen, Tested @ 77°F and 48 psig						
Thickness (mils)	30	30	30	30	30	30
Gas Transmission Rate, cm^3/24 hrs/$meter^2$/atmosphere	97.67	30.50	22.89	44.72	58.58	75.97
Permeability Coefficient in Barrers	1.13	0.35	0.26	0.52	0.67	0.88
70 hrs/212°F/ASTM #3 Oil						
Tensile Strength, psi	1870	1730	1390	1790	1580	1830
Modulus @ 200%E, psi	1130	1100	-	1100	1030	1550
Elongation, %	250	265	210	260	260	220
Hardness, Shore A	57	60	74	59	60	62
Volume Change, %	+44	+38	+44	+38	+42	+35
Compression Set, Method B, 70 hrs/212°F, %	16	49	36	32	36	16
Low Temperature Flexibility, ASTM D1043, Temp. where Young's Modulus is 10,000 psi, °F	-24	-6	-5	-13	-12	-18

TABLE III

EFFECT OF PLASTICIZER AND FILLERS ON THE NITROGEN GAS PERMEABILITY OF A LOW ACRYLONITRILE CONTENT BUTADIENE-ACRYLONITRILE RUBBER

	Parts by Weight							
Compounding Ingredients								
Paracril AJ	100	100	100	100	100	100	100	100
Stearic Acid	1	1	1	1	1	1	1	1
Zinc Oxide	5	5	5	5	5	5	5	5
Santocure	1	1	1	1	1	1	1	1
Sulfasan R	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3
Methyl Tuads	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3
TP-95 (Plasticizer)	--	20	20	20	20	20	20	20
FEF Carbon Black	50	50	50	50	50	50	50	50
325 Mesh Mica		--	50	--	--	--	--	--
50 Mesh Mica		--	--	50	--	--	--	--
Graphite		--	--	--	50	--	--	--
Magnesium Silicate		--	--	--	--	50	--	--
Dixie Clay		--	--	--	--	--	50	--
MT Carbon Black		--	--	--	--	--	--	50
Physical Properties								
Tensile Strength, psi	2240	1960	1760	1130	1280	1800	1800	1810
Modulus @ 200%E, psi	1180	610	900	610	760	680	750	910
Elongation, %	315	405	350	290	320	370	375	300
Hardness, Shore A	80	62	73	75	74	66	68	67
Permeability to Nitrogen Tested @ 77°F and 48 psig								
Thickness (mils)	30	30	30	30	30	30	30	30
Gas Transmission Rate, cm^3/24 hrs/meter^2/atmosphere	97.67	193.02	42.37	50.13	62.33	100.17	106.27	129.64
Permeability Coefficient in Barrers	1.13	2.42	0.49	0.60	0.75	1.21	1.27	1.59
70 hrs/212°F/ASTM #3 Oil								
Tensile Strength, psi	1870	1400	1610	940	970	1340	1390	1860
Modulus @ 200%E, psi	1130	630	660	550	680	490	700	900
Elongation, %	250	275	340	250	275	340	295	275
Hardness, Shore A	57	50	58	53	64	54	57	59
Volume Change, %	+44	+33	+36	+31	+29	+31	+34	+29
Compression Set, Method B, 70 hrs/212°F, %	16	16	64	57	26	33	30	18
Low Temperature Flexibility, ASTM D1043, Temp. where Young's Modulus is 10,000 psi, °F	-24	-49	-37	-38	-42	-40	-40	-40

TABLE IV

EFFECT OF FILLERS ON THE GAS PERMEABILITY OF A HIGH ACRYLONITRILE CONTENT BUTADIENE-ACRYLONITRILE RUBBER

	Parts by Weight					
Compounding Ingredients						
Paracril D	100	100	100	100	100	100
Stearic Acid	1	1	1	1	1	1
Zinc Oxide	5	5	5	5	5	5
Santocure	1	1	1	1	1	1
Sulfasan R	1.3	1.3	1.3	1.3	1.3	1.3
Methyl Tuads	1.3	1.3	1.3	1.3	1.3	1.3
FEF Carbon Black	50	50	50	50	50	50
325 Mesh Mica	--	50	--	--	--	--
Graphite	--	--	50	--	--	--
Magnesium Silicate	--	--	--	50	--	--
Dixie Clay	--	--	--	--	50	--
MT Carbon Black	--	--	--	--	--	50
Physical Properties						
Tensile Strength, psi	2690	1970	1700	2440	2590	2670
Modulus @ 200%E, psi	740	1150	1700	1600	1870	1720
Elongation, %	520	380	220	340	315	290
Hardness, Shore A	73	87	90	86	87	85
Permeability to Nitrogen, Tested @ 77°F and 48 psig						
Thickness (mils)	30	30	30	30	30	30
Gas Transmission Rate, cm^3/24 hrs/$meter^2$/atmosphere	12.54	11.37	8.62	12.26	12.80	12.51
Permeability Coefficient in Barrers	0.145	0.132	0.100	0.142	0.148	0.145
70 hrs/212°F/ASTM #3 Oil						
Tensile Strength, psi	2410	1710	1770	2520	2570	2700
Modulus @ 200%E, psi	710	1010	1580	1330	1740	1870
Elongation, %	435	340	260	365	335	265
Hardness, Shore A	70	83	90	85	86	86
Volume Change, %	[illegible]	+9	+4	+8	+7	+6
Compression Set, Method B, 70 hrs/212°F,	42	75	49	48	47	24
Low Temperature Flexibility, ASTM D1043, Temp. where Young's Modulus is 10,000 psi, °F	+26	+44	+46	+38	+38	+34

TABLE V

EFFECT OF FILLERS ON THE GAS PERMEABILITY OF POLYEPICHLOROHYDRIN RUBBER

	Parts by Weight						
Compounding Ingredients							
Hydrin 100	100	100	100	100	100	100	100
Zinc Stearate	1	1	1	1	1	1	1
Nickel Dibutyldithiocarbamate	1	1	1	1	1	1	1
Magnesium Oxide	5	5	5	5	5	5	5
NA-22	1.5	1.5	1.5	1.5	1.5	1.5	1.5
FEF Carbon Black	30	30	30	30	30	30	30
325 Mesh Mica	--	30	--	--	--	--	--
50 Mesh Mica	--	--	30	--	--	--	--
Graphite	--	--	--	30	--	--	--
Magnesium Silicate	--	--	--	--	30	--	--
Dixie Clay	--	--	--	--	--	30	--
MT Carbon Black	--	--	--	--	--	--	30
Physical Properties							
Tensile Strength, psi	1670	1090	1000	840	1190	1280	1160
Modulus @ 200%E, psi	480	1000	880	720	610	630	620
Elongation, %	540	415	435	365	530	535	560
Hardness, Shore A	64	78	79	80	75	75	74
Permeability to Nitrogen, Tested @ 77°F and 48 psig							
Thickness (mils)	31	32	32	31	31	32	32
Gas Transmission Rate, cm^3/24 hrs/meter^2/atmosphere	11.12	7.26	11.31	10.96	11.04	11.54	9.44
Permeability Coefficient in Barrers	0.133	0 087	0.136	0.115	0.132	0.142	0.116
70 hrs/212°F/ASTM #3 Oil							
Tensile Strength, psi	1310	1010	830	770	1020	1260	1160
Modulus @ 200%E, psi	490	900	650	700	680	880	790
Elongation, %	335	275	300	270	340	370	310
Hardness, Shore A	55	75	65	73	65	67	64
Volume Change, %	+15	+10	+9	+9	+15	+13	+11
Compression Set, Method B, 70 hrs/212°F, %	66	96	88	82	92	100	92
Low Temperature Flexibility, ASTM D1043, Temp. where Young's Modulus is 10,000 psi, °F	0	+10	+8	+20	+12	+8	+7

TABLE VI

EFFECT OF FILLERS ON THE GAS PERMEABILITY OF EPICHLOROHYDRIN/ETHYLENE OXIDE COPOLYMER RUBBER

	Parts by Weight						
Compounding Ingredients							
Hydrin 200	100	100	100	100	100	100	100
Zinc Stearate	1	1	1	1	1	1	1
Nickel Dibutyldithiocarbamate	1	1	1	1	1	1	1
Magnesium Oxide	5	5	5	5	5	5	5
NA-22	1.5	1.5	1.5	1.5	1.5	1.5	1.5
FEF Carbon Black	30	30	30	30	30	30	30
325 Mesh Mica	--	30	--	--	--	--	--
50 Mesh Mica	--	--	30	--	--	--	--
Graphite	--	--	--	30	--	--	--
Magnesium Silicate	--	--	--	--	30	--	--
Dixie Clay	--	--	--	--	--	30	--
MT Carbon Black	--	--	--	--	--	--	30
Physical Properties							
Tensile Strength, psi	1830	1310	1140	930	1370	1610	1800
Modulus @ 200%E, psi	1000	1180	1030	--	970	1400	1440
Elongation, %	350	255	245	210	290	280	255
Hardness, Shore A	70	83	83	85	78	81	79
Permeability to Nitrogen, Tested @ 77°F and 48 psig							
Thickness (mils)	30	30	30	30	30	30	30
Gas Transmission Rate, cm^3/24 hrs/$meter^2$/atmosphere	31.40	16.87	12.16	11.70	13.85	16.44	22.03
Permeability Coefficient in Barrers	0.401	0.214	0.141	0.135	0.162	0.190	0.256
70 hrs/212°F/ASTM #3 Oil							
Tensile Strength, psi	1680	1040	930	840	1210	1220	1310
Modulus @ 200%E, psi	930	990	920	--	920	870	710
Elongation, %	330	240	235	200	315	345	345
Hardness, Shore A	65	78	80	80	71	69	64
Volume Change, %	+11	+13	+12	+11	+14	+12	+13
Compression Set, Method B, 70 hrs/212°F, %	59	72	73	66	72	64	82
Low Temperature Flexibility, ASTM D1043, Temp. where Young's Modulus is 10,000 psi, °F	-34	-24	-23	-18	-28	-27	-31

TABLE VII

EFFECT OF PLASTICIZERS ON THE GAS PERMEABILITY OF EPICHLOROHYDRIN-ETHYLENE OXIDE COPOLYMER RUBBER

	Parts by Weight			
Compounding Ingredients				
Hydrin 200	100	100	100	100
Zinc Stearate	1	1	1	1
Nickel Dibutyldithiocarbamate	1	1	1	1
Magnesium Oxide	5	5	5	5
NA-22	1.5	1.5	1.5	1.5
FEF Carbon Black	30	30	30	30
TP-95	--	10	--	--
Diisoamyl Adipate	--	--	10	--
Alkaterge C	--	--	--	10
Physical Properties				
Tensile Strength, psi	1830	1440	1380	1760
Modulus @ 200%E, psi	1000	770	660	700
Elongation, %	350	325	340	445
Hardness, Shore A	70	67	64	70
Permeability to Nitrogen, Tested at 77°F and 48 psig				
Thickness (mils)	30	30	30	30
Gas Transmission Rate, cm^3/24 hrs/$meter^2$/atmosphere	31.40	71.62	92.17	58.59
Permeability Coefficient in Barrers	0.401	0.842	1.069	0.679
70 hrs/212°F/ASTM #3 Oil				
Tensile Strength, psi	1680	1330	1440	1670
Modulus @ 200%E, psi	930	770	660	740
Elongation, %	330	310	410	505
Hardness, Shore A	65	65	62	61
Volume Change, %	+11	+4	+4	+10
Compression Set, Method B, 70 hrs/212°F, %	59	56	67	86
Low Temperature Flexibility, ASTM D1043, Temp. where Young's Modulus is 10,000 psi, °F	-34	-44	-49	-34

CONCLUSIONS

The gas permeability of an oil-resistant elastomer can be significantly reduced by the addition of fillers of the proper size and shape without seriously affecting other physical properties.

Of the fillers evaluated, the platelike mica and graphite produced the greatest reduction in gas permeability.

Plasticizers increased the gas permeability of the elastomers evaluated in this study.

RECOMMENDATIONS

Those compounds developed under this project which possess low permeability to gases and are adequate with respect to performance at low temperatures and resistance to oils, fuels, and fluids should be evaluated as components in future weapon systems. Of particular interest would be the fluorosilicone rubber filled with mica and an epichlorohydrin/ethylene oxide copolymer rubber filled with FEF carbon black. These should be evaluated as the diaphragm in a hydraulic oil-nitrogen gas accumulator being developed for the XM150 cannon

Since the permeability of gases and vapors through a rubber film generally are similarly affected by changes in the composition of the rubber,[5] mica and graphite fillers used in this study to reduce gas permeability should be utilized to reduce the water-vapor permeability of rubber weapon components such as barrier bags for combustible-cased ammunition.

LITERATURE CITED

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